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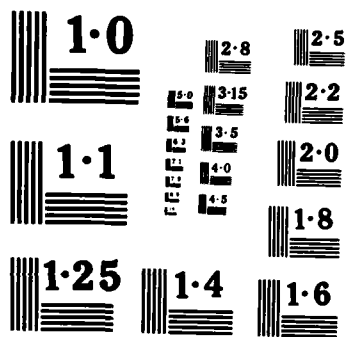
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## TECHNICAL REPORT NO. 1

TO

THE OFFICE OF NAVAL RESEARCH

CONTRACT No. N00014-85-K-0427, NR 412-019

THE COMBINED EFFECT OF STRESS STATE AND GRAIN SIZE  
ON HYDROGEN EMBRITTLEMENT OF TITANIUM

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1. REPORT NUMBER  No. 1	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle)  The Combined Effect of Stress State and Grain Size on Hydrogen Embrittlement of Titanium		5. TYPE OF REPORT & PERIOD COVERED
7. AUTHOR(s)  D. A. Gerard and D. A. Koss		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Metallurgical Engineering Michigan Technological University Houghton, Michigan 49931		8. CONTRACT OR GRANT NUMBER(s)  N00014-85-K-0427 NR412-019
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research 800 N. Quincy Street Arlington, Virginia 22217		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE July 1985
		13. NUMBER OF PAGES
		15. SECURITY CLASS. (of this report)  Unclassified
16. DISTRIBUTION STATEMENT (of this Report)  Distribution of this document is unlimited		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)  Hydrogen Embrittlement, Titanium, Multiaxial States of Stress, Grain Size		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  The influence of grain size on the hydrogen embrittlement of titanium sheets is shown to depend on stress state. Results are presented which indicate that the deleterious effect of a large grain size on the susceptibility to hydrogen embrittlement is more severe in equibiaxial tension than in uniaxial tension. This effect may be understood in terms of the sensitivity of both void nucleation (due to hydride fracture) and void link-up due to the combined effects of large grain/hydride sizes and the large multidirectional, normal stresses that develop during deformation of plastically		

20. anisotropic sheets in equibiaxial tension.

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# THE COMBINED EFFECT OF STRESS STATE AND GRAIN SIZE ON HYDROGEN EMBRITTLEMENT OF TITANIUM

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## Introduction

Commercially pure titanium is very resistant to embrittlement due to hydrogen when tested in the form of fine-grained specimens at low-to-moderate strain rates in uniaxial tension.<sup>1-3</sup> It is well known that Ti becomes susceptible to hydrogen embrittlement in the presence of a notch, at low temperatures or high strain rates, or large grain sizes.<sup>1-6</sup> Recent studies have also indicated a sensitivity to stress state with Ti sheets exhibiting pronounced hydrogen embrittlement in equibiaxial tension even though the loss of ductility in uniaxial tension is negligible.<sup>7,8</sup> The purpose of this communication is to explore the combined effects of two of the above factors, grain size and stress state, on the hydrogen embrittlement of Ti. Specifically, we shall show that the adverse effect of a large grain size on the susceptibility to hydrogen embrittlement is more severe in equibiaxial tension than in uniaxial tension.

## Experimental Procedure

Commercially pure Ti sheets (Ti-50A) containing 0.130 wt % oxygen were heat treated to produce two levels of grain sizes and hydrogen contents. In all cases, the sheets received an anneal at 700°C followed by a helium gas quench. For two groups of specimens, the anneal was performed in a dynamic vacuum of  $\sim 1.3 \times 10^{-6}$  KPa which resulted in hydrogen content of 25 wt ppm. Two other groups of specimens were also annealed at 700°C but in hydrogen gas to

permit charging to a level of 1850 wt ppm. Prior to the above heat treatments, one batch of specimens from each of the above two groups received a 9% cold roll reduction in sheet thickness from .81 to .74mm. This caused grain growth during the subsequent 700°C anneals from an initial average 0.015 mm grain size to an average size of 0.030 mm; the grains remained equiaxed. Thus, specimens with two grain sizes, 0.015 and 0.030 mm, and two hydrogen contents, 25 and 1850 wt ppm H, were prepared.

The microstructures of both the uncharged and charged specimens are shown in Fig 1. Fig. 1 c and d indicate the presence of both intergranular and intragranular hydrides in the charged specimens. It is important to note that the coarse grain structure permits larger hydrides to form; for example, optical microscopy indicates a maximum hydride size of  $\sim 100\mu\text{m}$  in the coarse grain material compared with hydrides up to  $\sim 40\mu\text{m}$  in size in the fine-grained material. Grain sizes are sufficiently small so that even in the coarse-grain material, there are  $> 24$  grains through a sheet specimen thickness.

Mechanical testing followed the procedure of Bourcier and Koss<sup>7,8</sup> in which equibiaxial testing is accomplished by punch-stretch testing of gridded sheet specimens at an average equivalent strain rate of  $3 \times 10^{-3} \text{ s}^{-1}$ . Uniaxial tests were also performed on specimens with  $\sim 1\text{mm}$  grids in order to permit determination of the plastic anisotropy as well as fracture strains adjacent to and including the fracture surface. It should be noted that the sheets are plastically anisotropic with  $R \approx 2$  and  $P \approx 3$  to 6, where R and P are  $\epsilon_{\text{width}}/\epsilon_{\text{thickness}}$  in tensile tests parallel to either the rolling direction (R) or the transverse direction (P).

### Experimental Results and Discussion

The combined influence of grain size and stress state on the hydrogen embrittlement of Ti sheet is shown in Fig. 2. Before discussing its implications, we wish to comment on certain experimental aspects of Fig. 2. The Figure is based on measurements of the major and minor principal strains of an  $\sim 1\text{mm}$  element spanning the fracture surface. The calculation of the local equivalent strain to fracture  $\bar{\epsilon}_f$  uses the previously reported R and P values and an assumption of Hill's original, quadratic yield criterion<sup>9</sup>; this has been shown to provide an adequate description of the multiaxial stress-strain behavior of Ti containing hydrogen.<sup>10</sup> Since the macroscopic fracture path in equibiaxial tension is roughly parallel to the rolling direction of the sheet, the ratios in Fig. 2 are based on uniaxial data from specimens tested normal to the rolling direction (or parallel to the transverse direction).

Fig. 2 compares the ratios of fracture strain in equibiaxial tension to those in uniaxial tension for two levels of grain size and hydrogen content. Fig. 2 shows that there is very little, if any, effect of grain size on the ratio of fracture strain in equibiaxial tension to that in uniaxial tension for the uncharged Ti. In contrast, the loss of ductility in equibiaxial tension for the hydrogen-charged sheets is much more pronounced for the coarse-grain material than in the finer grained counterparts. Thus, the data in Fig. 2 indicate that the hydrogen embrittlement of Ti sheet at room temperature is most severe in coarse-grain material when subjected to equibiaxial tension.

That increasing either grain size (in uniaxial tension) or biaxiality of stress state (at constant grain size) increases the severity of hydrogen



embrittlement in hydride-forming alloys is known.<sup>2,3,7,8</sup> Thus it is not entirely surprising that the stress-state effect is itself sensitive to grain size. The cause of this effect may be understood in the context of the previous studies, the present metallographic/fractographic observations, and the void nucleation/void link-up steps of the ductile fracture process. Grain-size effects in uniaxial tension have been previously attributed to an increased hydride plate size associated with the coarser grains.<sup>2,3</sup> This is consistent with present observations of  $<100\text{ }\mu\text{m}$  hydrides in the coarse-grain sheets but  $<40\text{ }\mu\text{m}$  hydrides in the fine-grain material. The combination of large hydrides and large normal stresses required to deform the plastically anisotropic sheets in equibiaxial tension should decrease the strain to fracture of the hydrides, thus enhancing void nucleation at smaller strains. In addition, the presence of large, interconnected hydrides also assist void link-up by providing paths of easy fracture along the flat hydride faces,<sup>8,11</sup> especially under the multidirectionality of the two major principal stresses in equibiaxial tension. This effect is apparent in Fig. 3 in which the fracture surfaces of the coarse-grain material show faceting due to void link-up along the hydride plate faces in both uniaxial and equibiaxial tension. As shown in Fig. 3, such faceting is most pronounced in the coarse-grain material tested in equibiaxial tension, indicating the enhancement of void link-up when both large, interconnected hydrides and a multidirectional tensile stress state are present.

In view of the above, we may summarize. Increasing grain size increases the susceptibility of Ti (and probably other hydride-forming alloys) to hydrogen embrittlement at high degrees of stress biaxiality/triaxiality. This effect appears to be a consequence of an enhancement of both void nucleation

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(due to hydride fracture) and void link-up at large grain sizes/biaxial stresses. Void nucleation should be enhanced as the large grains create conditions for large hydrides<sup>2,3</sup> which in turn fracture and form voids at smaller strains at the large normal stresses required to deform plastically anisotropic ( $R$  and  $P > 1$ ) sheets in equibiaxial tension.<sup>6</sup> Void link-up should also be enhanced as the large interconnected and plate-like hydrides create paths for especially easy void link-up when subjected to the multidirectional major principal stresses in equibiaxial tension. These effects should be even more pronounced under triaxial states of stress, such as near notches or cracks, provided that the stress state is sufficiently long range to encompass several large grains/hydrides, thus permitting enhancement of both void nucleation and link-up.

#### Acknowledgements

This research was supported by the Office of Naval Research through Contract No. N00014-85-K-0427, NR 412-019.

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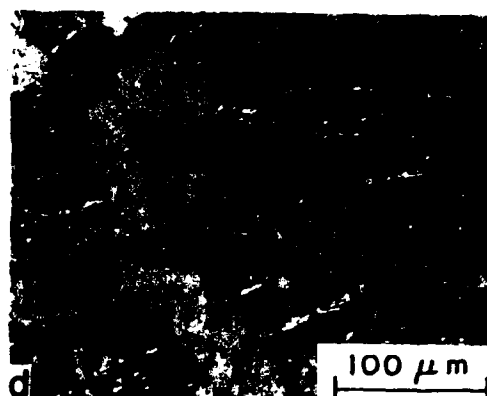
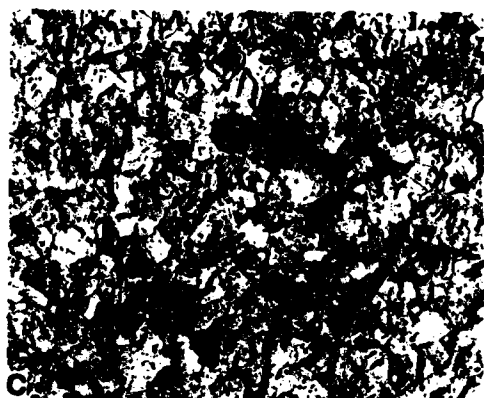
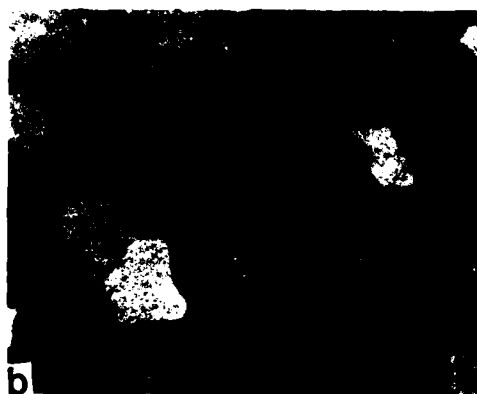


Fig. 1. Optical micrographs showing fine-grain (a and c) and coarse-grained (b and d) titanium containing either 25 or 1850 (c and d) wt ppm hydrogen.

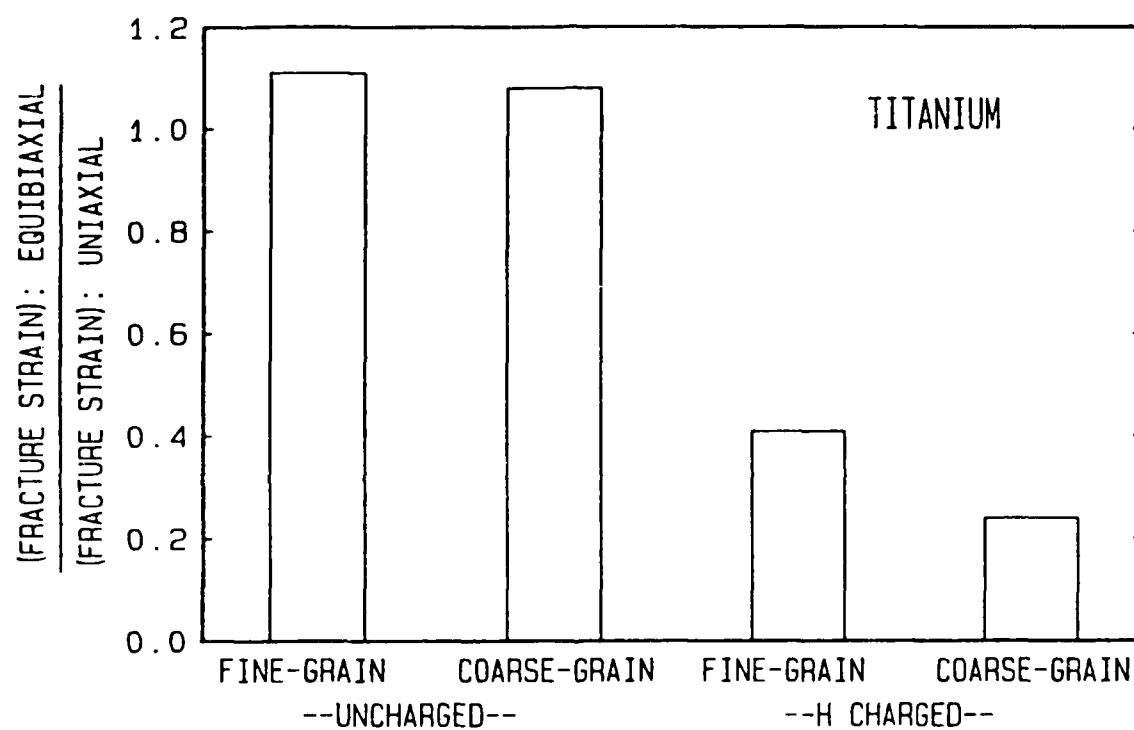


Fig. 2. The ratio of Hill equivalent strain to fracture in equibiaxial tension to that in uniaxial tension for Ti with either 0.015 or 0.030mm grain size and 25 or 1850 wt ppm H.

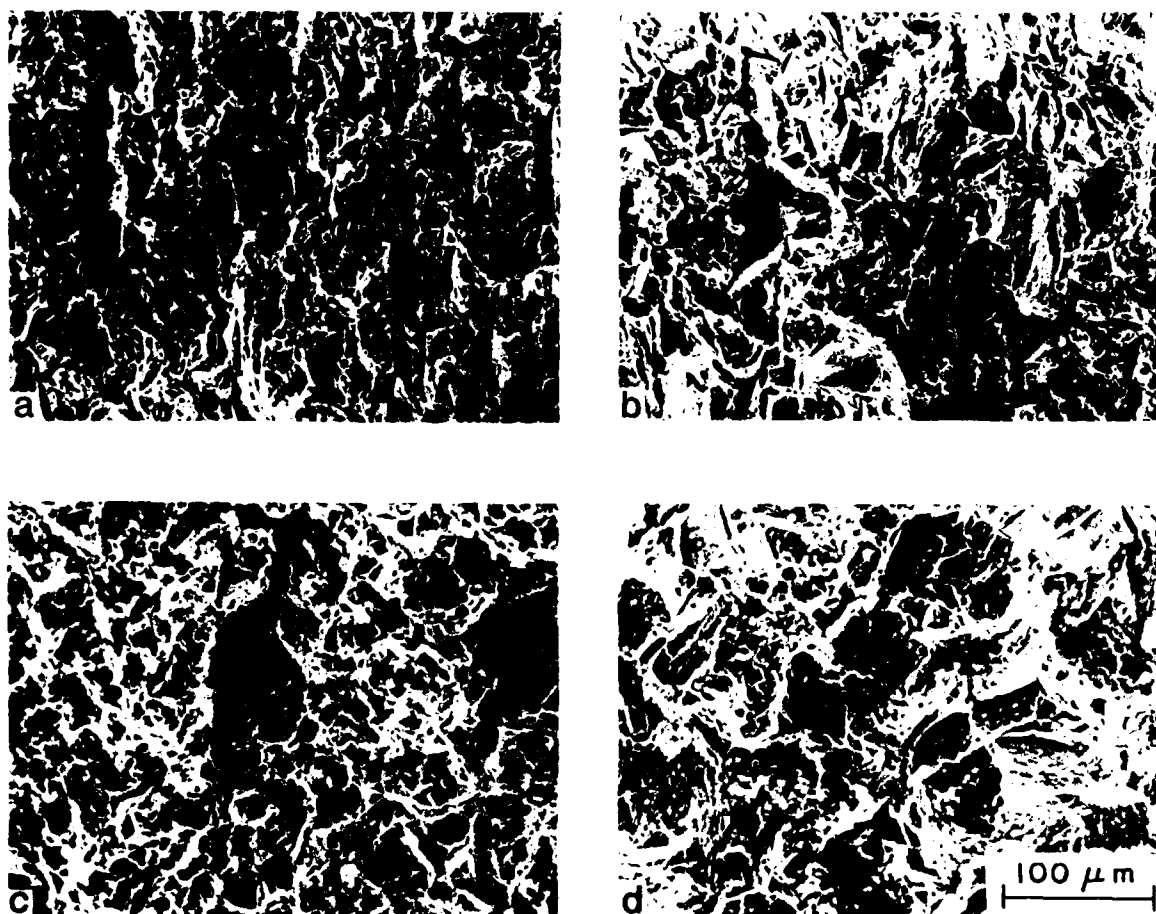


Fig. 3. Scanning electron fractographs of titanium containing 1850 wt ppm hydrogen and tested in uniaxial tension (a and b) or equibiaxial tension (c and d). Micrographs (a) and (c) are of fine-grain material while (b) and (d) are of coarse-grained sheets.

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